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MEASUREMENT OF ELASTIC MODULUS OF ALUMINA AND BARIUM STRONTIUM TITANATE WAFERS PRODUCED BY TAPE CASTING METHOD

Shri N. Singh

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Munitions Engineering Technology Center

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Elastic modulus measurements were made using a three-point configuration testing method. Samples of barium strontium titanate (BST) were made using a regular powder pressing, sintering, pelletizing, and sawing method (pelletizing method), as well as a tape casting technique. Samples of aluminum oxide (alumina) were made by a tape casting method.

For BST, a value of elastic modulus between 99 to 114 GPa was obtained for the pelletizing method and 161 GPa for the tape casting BST material. For the alumina, an average value of elastic modulus of 249 GPa was measured.

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INTRODUCTION

During a recent (2007 to 2011) program, there existed a need for a modeling and simulation study to determine the stresses inside a device fabricated using thin wafers of barium strontium titanate (BST) and aluminum oxide (alumina) ceramic during launch of a system. Sandia National Laboratory, Livermore, California was given the task of modeling and simulation. For such a simulation, the values of Young's modulus for alumina and BST were essential requirements.

LITERATURE SEARCH

Mechanical properties of BST are very scarce and no data dealing with mechanical properties were found. However, since barium and strontium make a complete solid solution, the properties of barium titanate (BT) will be similar to those of BST. Therefore, the literature for values for BT was searched. Pohanka and Smith (ref. 1) listed Young's modulus for BT in J/m^2 at 4 at 25°C and 2.5 at 150°C. Another work by Scholz, Schneider et al. (ref. 2) lists Young's modulus for BT at 181 GPa. A.A. Wereszczak, L Riester et al. (ref. 3) find BT multilayer capacitors in Young's modulus at 193 GPa. It seems that there is a close agreement between these three studies.

Mechanical properties of alumina have been determined by several researchers and are listed in the literature. A value of Young's modulus at 390 GPa is listed in Fundamentals of Ceramics (ref. 4). "Mechanical Properties of Ceramics" lists the value of Young's modulus for alumina at 297 GPa. Another study of modulus of alumina as a function of its density lists the values between 260 to 410 GPa depending on its density (ref. 5). There is a decently large variation between these two values. It is to be noted that sample preparation, impurity levels, and sample size may be the reason for such variations.

It is worth mentioning that most of the samples are prepared by ball milling powders, calcining, crushing, milling, isostating pressing, and forming the sintered products that are then used to mechanically cut and polish to then obtain the samples for measurements.

Because there was a large variation in the published data and since samples of both BST and alumina were readily available to us, it was decided to perform the measurements of elastic modulus to confirm and verify the values found in the published literature. These measurements, done on actual materials, will also provide the most reliable data for the purpose of the experiment.

MEASUREMENTS OF ELASTIC MODULUS

Two kinds of samples were used for BST and one kind for alumina. For BST, the first samples were obtained from the U.S. Army Research Laboratory (ARL), Aberdeen, MD, and were produced by a standard method using powders of barium and strontium carbonates. The steps followed are summarized below

- Mix stoichiometric amounts of barium carbonate, strontium carbonate, and titanium oxide together.
- Powder pressed into a 32-mm diameter pellet using auniaxial press.
- Pellet heated in air for 10 hr to 1100°C.
- Pellet reground into a fine powder with a mortar and pestle.
- Powder isostatically pressed into a 32-mm diameter pellet with a pressure of 45 to 50 ksi.
- Pellet heated to 1500°C in air for 25 hr.

This resulted in sintered pellets that were cut using fine diamond-coated saws to prepare wafers of 25 by 8 by 0.5 mm.

The other samples of BST wafers, as well as alumina wafers, were produced by a technique known as tape casting. Tape casting is widely used in the production of paper, plastic, and paint, as well as in the production of thin ceramics sheets. The basic principles of casting are described on the flow chart depicted in figure1. The ceramic powders are added in the ball mill along with deflocculants/dispersants. The mixture is then ball milled until the proper dispersion of solid particles in the solvent is achieved. The next step consists of mixing the plasticizers and binders into the slurry produced in the ball mill. De-airing, usually by vacuum, is necessary to remove the entrapped air. This is accomplished in a simple device such as a desiccator. For the most critical applications, such as very thin tapes or very smooth thin film substrates, a slip filtration step is usually needed just before the casting step. The details of tape casting can be found in reference 6, as well as in a technical report (ref. 7).

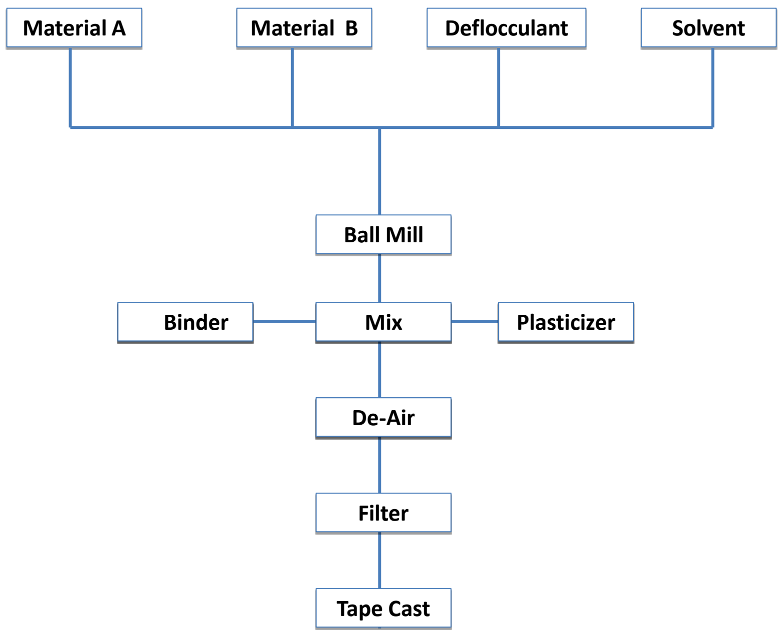


Figure 1
Schematic of tape casting method (ref. 6)

The finished sintered wafers of BST and alumina are shown in figure 2.

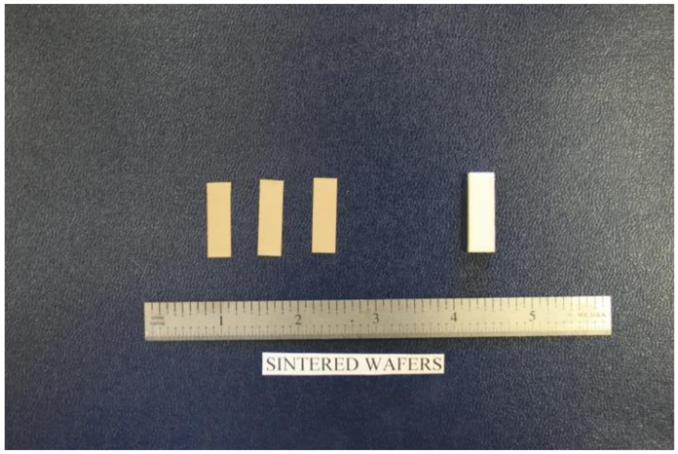


Figure 2
Wafers of tape cast BST (dark color) and alumina (light color)

The most common method of fixture testing, the three-point configuration test, was used to determine the elastic modulus. The measurements were made at Sandia National Laboratories, Livermore, California. Figure 3 shows the experimental setup and the magnified view of the three-point test. The formula used to calculate the modulus is indicated at the right hand corner of figure 3. The dimensions of the wafers are explained in the diagram.

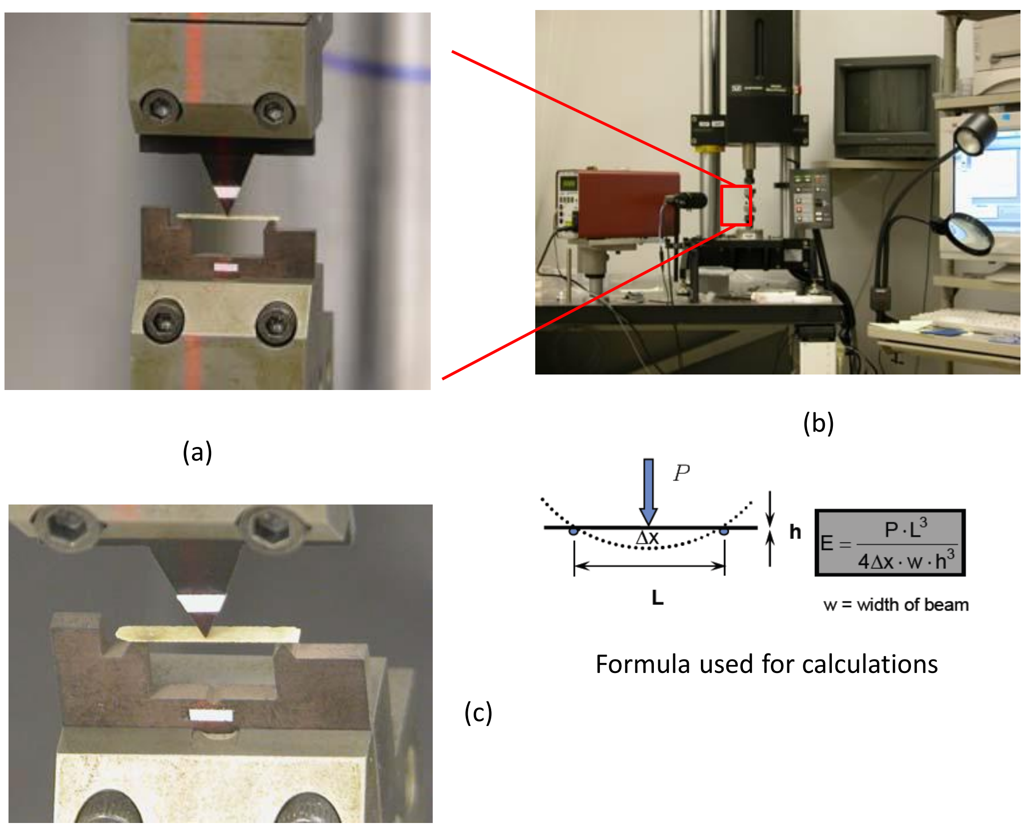
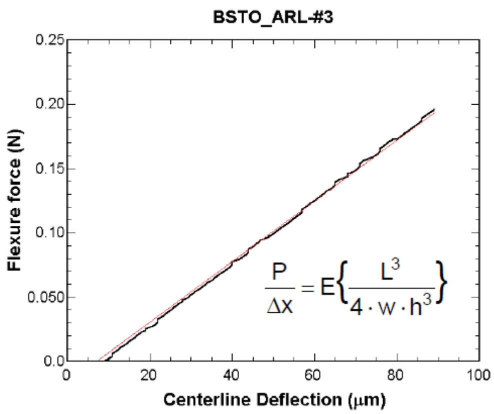


Figure 3
The three-point test equipment (a, b, and c) and formula used for calculations

The details of the measurements and the results are shown in figure 4 and summarized in figure 5. These measurements were made on the samples produced at ARL and by the pelletizing method described previously. An average value of the elastic modulus of around 105 GPa was calculated using several samples as described in figure 5.

Modulus measurement- multiple tests on single beam



	Modulus (GPa)	Max Load (mN)	Max Stress (MPa)	Max CL disp (um)
1	-	124.72	37.93	49.5
2	104.45	197.67	60.12	89.8
3	104.03	195.95	59.6	89.4
4	104.65	195.48	59.46	89.4
5	104.58	197.68	60.12	90.4
6	104.76	197.03	59.93	89.8
7	105.64	194.45	59.14	89.5
8	103.98	196.55	59.78	90.6
9	104.02	196.3	59.7	90.4
*10	104.43	229.3	***70.01**	105.22
Mean	104.5	169.63	58.58	105.2
Standard Deviation	0.51	63.58	7.95	63.9
Minimum	103.98	0.43	37.93	49.5
Maximum	105.64	197.68	70.01	283.5
Range	1.65	197.25	32.08	233.9

*Test 10: beam taken to failure

- For any beam, mod. measurements are consistent
- 104-105 GPa modulus has been measured in 1st beam tested.
- Beam flexed to failure:
  - Failure stress = 70 MPa

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Figure 4
Multiple tests measurements on a single beam of wafer from ARL material of BST

Summary

➢Modulus differences between samples are likely the result of:
- Dimensional variability in machining
- Local porosity and defects

	Sample #		
	2	3	4*
Avg Mod (GPa)	**99.3**	**104.5**	**113.9**
Std Dev	1.2	0.3	0.8
Max	100.3	104.8	114.6
Min	97.6	104.0	112.7

* 4 measurements only

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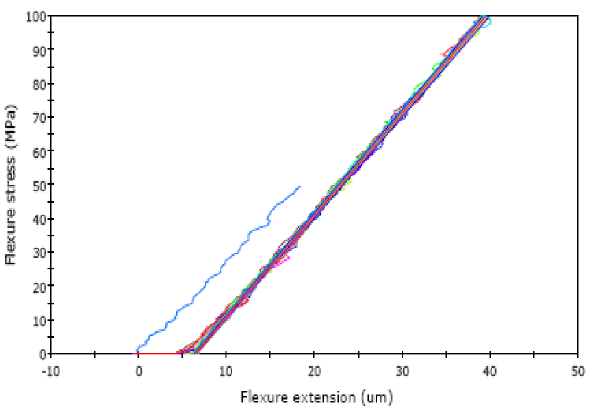
Figure 5
Summary of measurements performed on 3 different samples of wafers from ARL

The measurements made for the BST wafers produced by the tape casting method are shown in the figure 6. Samples of two different wafer thicknesses (0.5 and 0.3 mm) were used, and details of the measurements and the results are shown in figure 6. Results of many tests using both thicknesses show very consistent values of 161 GPa.

Results –tape cast BST wafers



- Modulus
  - –0.5-mm thick (0.47)
    - •161.4 +/- 0.6 GPa
    - •165.5 +/- 1.8
  - –0.3-mm beams (0.28)
    - •163.6 +/- 1.1 GPa
    - •157.9 +/- 0.9
- Failure flexure stress
  - – two beams at 0.3 mm
    - •98/115 MPa

	Specimen label	Flex Modulus (GPa)	Maximum Load (mN)	Maximum Stress (MPa)	Max Cl disp (um)
1	BST Thick #1-1	-----	1174.26	49.57	18.42
2	BST Thick #1-2	-----	1880.51	79.38	33.18
3	BST Thick #1-3	161.16	2368.94	100.00	39.38
4	BST Thick #1-4	161.62	2366.71	99.90	38.92
5	BST Thick #1-5	161.37	2365.33	99.84	39.71
6	BST Thick #1-6	161.54	2364.46	99.81	39.55
7	BST Thick #1-7	162.21	2363.63	99.77	39.22
8	BST Thick #1-8	160.26	2362.49	99.72	39.54
9	BST Thick #1-9	161.88	2361.32	99.67	38.67
Mean		161.43	2178.63	91.96	36.29
Standard Deviation		0.61933	409.27296	17.27603	7.00680
Minimum		160.26	1174.26	49.57	18.42
Maximum		162.21	2368.94	100.00	39.71
Range		1.95	1194.69	50.43	21.30

Figure 6
Summary of measurements made on BST wafers produced by tape casting method

The values of modulus for the tape cast material is significantly higher than the ones made by the standard powder pressing, calcining, grinding, and pelletizing method. The wafers made by this method were less uniform as compared to the tape cast material. It is also to be noted that the study of the microstructure had shown that there was more porosity in samples made at ARL with the standard method. On the other hand, the wafers made by tape casting were more uniform in thickness and had more uniformly distributed porosity of a smaller size. Although the value of 161 GPa is different from the value for BT, it is not too far from those 161 versus180 to 190 GPa. It is to be noted that these values are for different materials, and such variation is expected.

Measurements of elastic modulus for alumina were done on the wafers produced by the tape casting method. Thickness of the wafer was 0.5 mm. The thicker wafers measuring 1 mm were too thick for the measurements. The results are shown in figure 7. Three values are listed as 245.2, 249.6, and 253.2 yielding an average value of 249.3 GPa.

Measurements done on Al_2O_3 wafers

- Density of Al_2O_3 (99.9%) ≈ 3.96 g/cm^3
  - Estimated density of coupon: 3.62 g/cm^3
    - Relative density = 91.4%
  - Modulus at 100% → 400 GPa
  - Modulus at 91% → < 300 GPa
- 0.5-mm thick beams
  - 245.2 +/- 1.4 GPa
  - 249.6 +/- 3.12 GPa
  - 253.2 +/- 3.7 GPa
- 1.0-mm beams: probably too stiff for good measurements
  - Generally in the 230 to 250 GPa range

Grade	Al_2O_3/Porosity %	Young's modulus GPa
A1	≥ 99.6 / 0 - 2	410 - 380
A2	≥ 99.8 / < 1	405 - 380
A3	≥ 99.5 / < 1	400 - 398
A4	≥ 99.6 / 3 - 6	380 - 340
A5	≥ 99.0 / 1 - 5	380 - 340
A6	96.5-99.0 / 1 - 5	375 - 340
A7	94.5-96.5 / 1 - 5	370 - 300
A8	86.0-94.5 / 2 - 5	330 - 260
A9	80.0-86.0 / 3 - 6	330 - 260

**VTT manufacturing, 1996, ISBN 951-38-4987-2*

10-Feb-14 SHG, 8758

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Figure 7
Modulus measurements on 0.5-mm wafers of alumina data on the table from the literature

RESULTS AND DISCUSSION

Elastic modulus measurements were made using the three-point configuration testing method. Samples of BST were made using a regular powder pressing, sintering, pelletizing, and sawing method (pelletizing method), as well as a tape casting technique. Samples of alumina were made by tape casting method.

For BST, a value of elastic modulus of between 99 to 114 GPa was obtained for the ARL method and 161 GPa for the tape casting BST material. This is a result of wafers of BST made by tpe casting having less porosity that the other method. Also noted, the variation between the measured values for the tape cast samples was very little compared to those from the pelletizing method. This is due to the fact that the wafers of the tape cast method were found to be more uniform in thickness than those made by the other method (using powder, calcining, sintering, making pellets, and then cutting into wafers).

For the alumina, an average value of elastic modulus of 249 GPa was measured. This value is lower than reported in the literature and seems to be related to the relative density. The density of pure alumina should be 3.96 g/cm^3. However, the coupon was tested to have a density of about 3.62

g/cm^3 indicating a high value of porosity resulting in a relative density of 91.4%. According to the chart in figure 7, the values obtained at this relative density correlate well with what was reported in the literature. The high value of porosity may be an indication of insufficient sintering process i.e., either less time or low sintering temperature.

CONCLUSIONS/RECOMMENDATIONS

Toward the end of the program, the plan was to make measurements of yield strength, ultimate tensile strength, and elongation of the barium strontium titanate (BST) and aluminum oxide (alumina). Samples of these materials could be easily made into dog bone tensile samples using a tape casting method. However, due to lack of time, those measurements were not completed. It is recommended that when and if such measurements are required, the tape casting method will be most suited for producing the samples.

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